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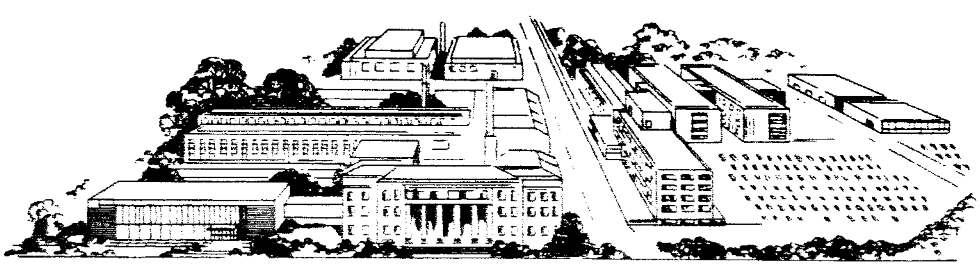

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FIRST QUARTERLY PROGRESS REPORT

on

IMPROVEMENT OF CREEP STRENGTH AND LOW-TEMPERATURE DUCTILITY OF REFRACTORY METALS BY MEANS OF MECHANICAL TWINNING

to

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

November 30, 1964

by

J. W. Edington

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January 29, 1965

National Aeronautics and Space Administration
Office of Research Grants and Contracts
Office of Space Sciences
600 Independence Boulevard
Washington, D. C. 20546

Attention Mr. T.L.K. Smull

Gentlemen:

Contract No. NASr-100(05), "Improvement of Creep Strength and Low-Temperature Ductility of Refractory Metals by Means of Mechanical Twinning"

Enclosed are 25 copies of the First Quarterly Report covering the contract period September 1 to November 30, 1964.

Sincerely yours,

J. W. Edington

J. W. Edington
Metal Science Group

JWE:cm
Enc. (25)

cc: Mr. J. Maltz (2)
Mr. W. D. Klopp (1)

IMPROVEMENT OF CREEP STRENGTH AND LOW-TEMPERATURE DUCTILITY OF REFRACTORY METALS BY MEANS OF MECHANICAL TWINNING

by

J. W. Edington

INTRODUCTION

It has been shown previously by Tardiff et al.[1] that there is an increase in micro hardness with increasing twin density, and Reid[2] has shown that deformation twins harden single crystals of columbium more than do slip dislocations.

Furthermore, experiments have shown that mechanically twinned structures are more stable at high temperatures than slipped structures[3] and that the presence of mechanical twins can increase the recrystallization temperatures of the cold-worked metal[4]. Finally, work currently in progress[5] shows that the prior existence of twins in polycrystalline iron can prevent cleavage-crack propagation and so lower the ductile-brittle transition temperature.

Thus, it is conceivable that twins in bcc metals may improve their mechanical properties at both high and low temperatures. This report describes some preliminary investigations of the possible mechanical-property improvements outlined above.

EXPERIMENTAL WORK

The materials chosen for these investigations were a columbium-40 wt % vanadium alloy, columbium, and tungsten. The columbium alloy was chosen for an evaluation of the high-temperature strengthening mechanism because it is known that closely spaced twins can be introduced by deformation at room temperature. The columbium and tungsten were used to evaluate the possible improvement of ductility at low temperatures, since a comparison of the behavior of materials known to behave in a ductile and in a brittle manner was considered to be important.

The columbium alloy was produced as a drop casting after melting columbium and vanadium in a nonconsumable-electrode electric-arc furnace. The columbium and tungsten were produced industrially in rod form. The columbium, tungsten, and alloy constituents were commercially pure. An exact analysis is not available at present.

The columbium alloy proved to be ductile at room temperature. After an initial 10% reduction in area, by rod rolling and annealing for one hour at 1400 C, the material was rolled to 0.050-inch thickness before a final annealing treatment of 1 hour at 1300 C.

RESULTS AND DISCUSSION

High-Temperature Strengthening

Previous work by Reid[2] has shown that mechanical twins give rise to a high-temperature strengthening in single crystals of columbium. The first step of this investigation was to evaluate the effect in polycrystalline material. For this work, polycrystalline columbium specimens were used. Two specimens were prestrained to 1% elongation at -196 and 30 C so that one specimen contained deformation twins and slip dislocations, while the other contained only slip dislocations. These specimens were tested in tension at 600 C. The stress-strain curves are shown in Figure 1. It will be noted that there is no real difference in the mechanical properties of the two specimens. However, it is well known that the yield stress of columbium is relatively unaffected by changes in grain size, and so if deformation twins strengthen material in the same way as grain-size refinement, their strengthening effect would be expected to be similarly inconsequential in this material.

Consequently, tensile tests were carried out on a polycrystalline molybdenum-50 wt % rhenium alloy. Two mechanical tests were carried out. Specimen A was strained to 1% elongation at room temperature, then tested at 600 C. Specimen B was tested in the as-annealed condition at 600 C. The stress-strain curves are reproduced in Figure 2. It can be seen that, after equivalent total elongations, the flow stress of Specimen A was $\simeq$30% higher than that of Specimen B. Both the ultimate tensile strength and elongation to fracture of Specimen A were slightly greater than those properties of Specimen B. These tests, therefore, show that twin strengthening occurs both in single crystals of columbium and in the polycrystalline molybdenum-50 wt % rhenium alloy but not in polycrystalline columbium.

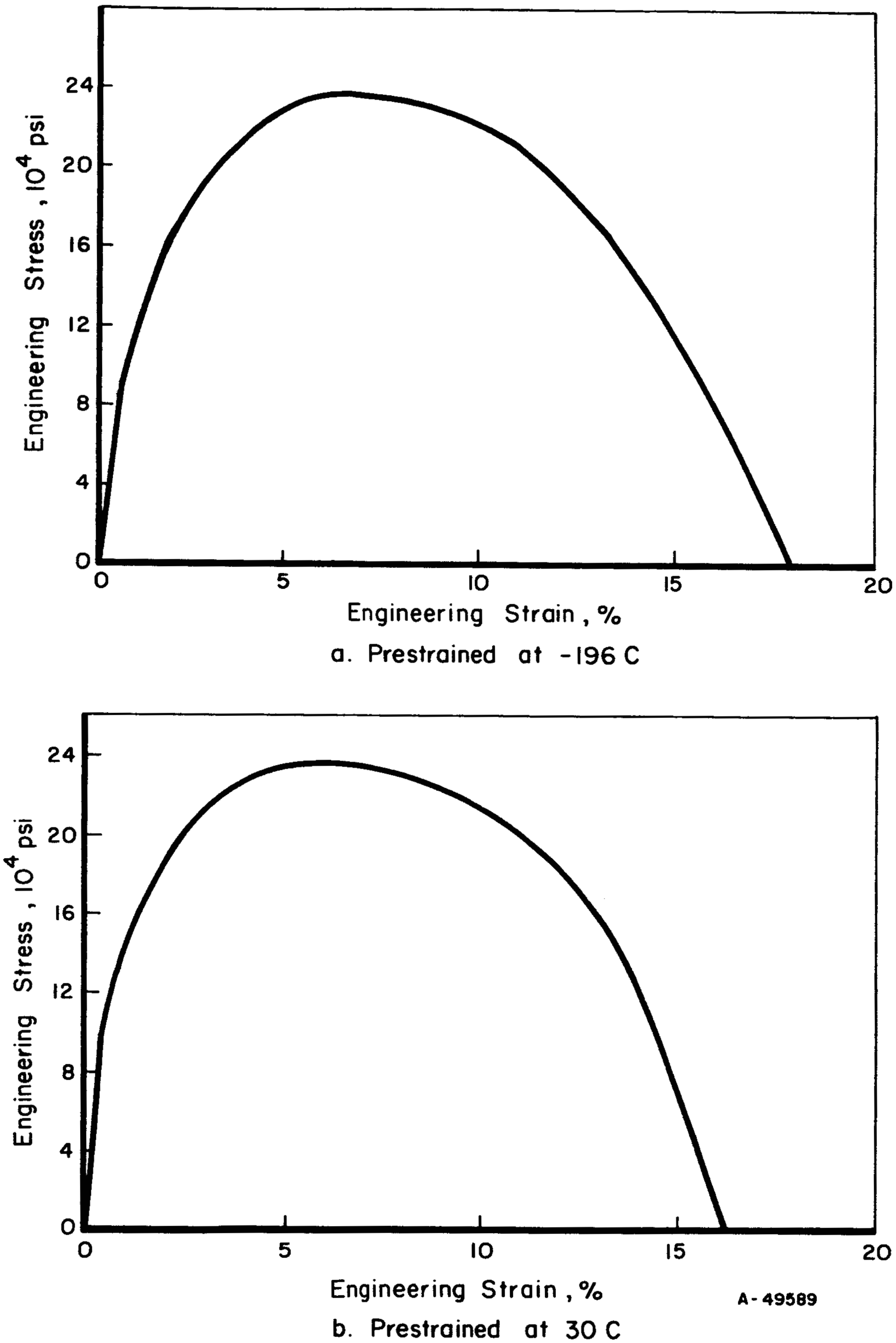


FIGURE 1. THE STRESS-STRAIN CURVE FOR POLYCRYSTALLINE COLUMBIUM SPECIMENS TESTED IN TENSION AT 600 C IN AIR

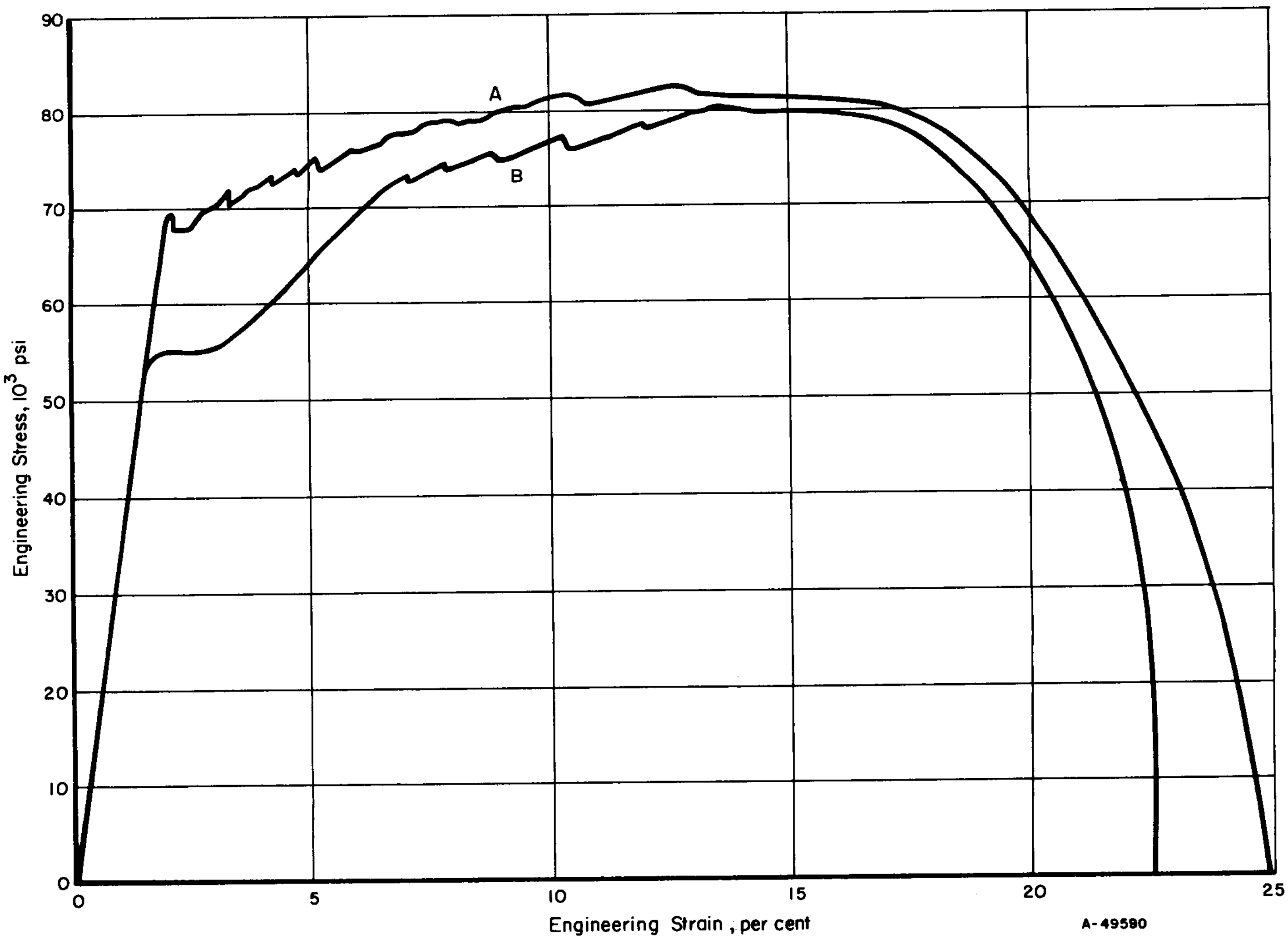


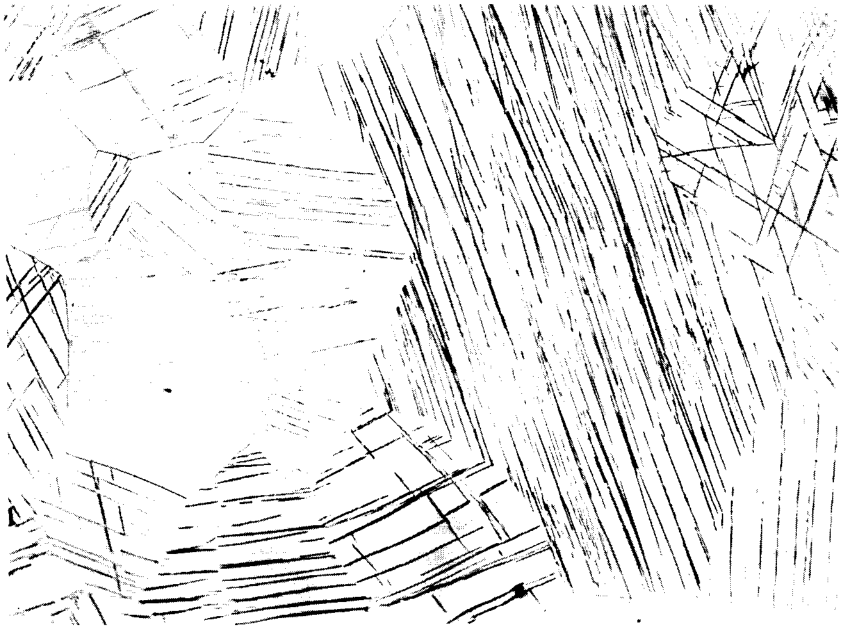
FIGURE 2. THE STRESS-STRAIN CURVES OF MOLYBDENUM-50 WT% RHENIUM

Specimen A – prestrained to 1% elongation at 30 C
Specimen B – as annealed

Twin Stability at High Temperatures

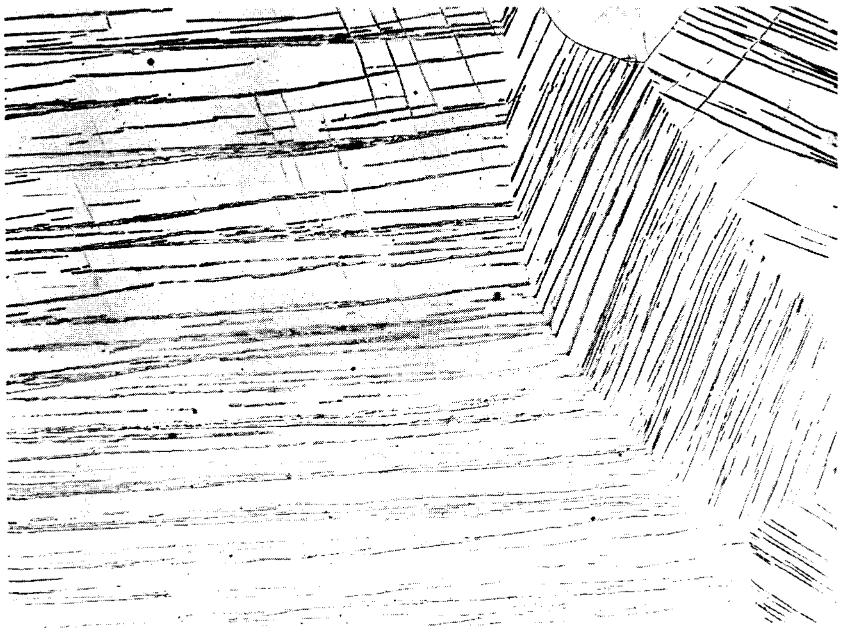
After the initial investigation described above, it was necessary to determine the annealing characteristics of the mechanically twinned columbium-40 wt % vanadium alloy which was to be used for most of the investigation. A series of strip specimens 0.050 inch thick were made as described previously, rolled to 5% reduction in thickness at room temperature, and annealed for 1 hour at temperatures between 900 and 1300 C. Figure 3 shows typical areas from some of the specimens. Pinching off of the twins at incoherent twin boundaries begins to occur at 1000 C, and the lengths of twins appear to be continually decreased during annealing at higher temperatures until at 1300 C, recrystallization occurs. Even after annealing at 1200 C, a few islands of twins remain.

The results of microhardness measurements carried out on these specimens are shown in Figure 4. It can be seen that there is a rapid softening of the material between 1000 and 1100 C, when the twins begin to anneal out. Surprisingly there is no correlation in hardness with twin density within grains in specimens annealed at 1100 and 1200 C. It is also surprising to note that there is no difference in hardness between the partly and fully recrystallized material, i.e., that annealed at 1100 C and 1300 C, respectively. However, hardness is not a very sensitive measure of mechanical strength.



100X 16633

a. As Rolled.



100X 16634

b. Annealed 1000 C.

FIGURE 3. PHOTOMICROGRAPHS SHOWING THE EFFECT OF ANNEALING TEMPERATURE ON THE MICROSTRUCTURE OF COLUMBIUM-40 WT% VANADIUM ALLOY PREVIOUSLY ROLLED TO 5% REDUCTION IN THICKNESS AT 30 C

100X 16636

c. Annealed 1100 C.

100X 16637

d. Annealed 1200 C.

100X 16638

e. Annealed 1300 C.

FIGURE 3. (CONTINUED)

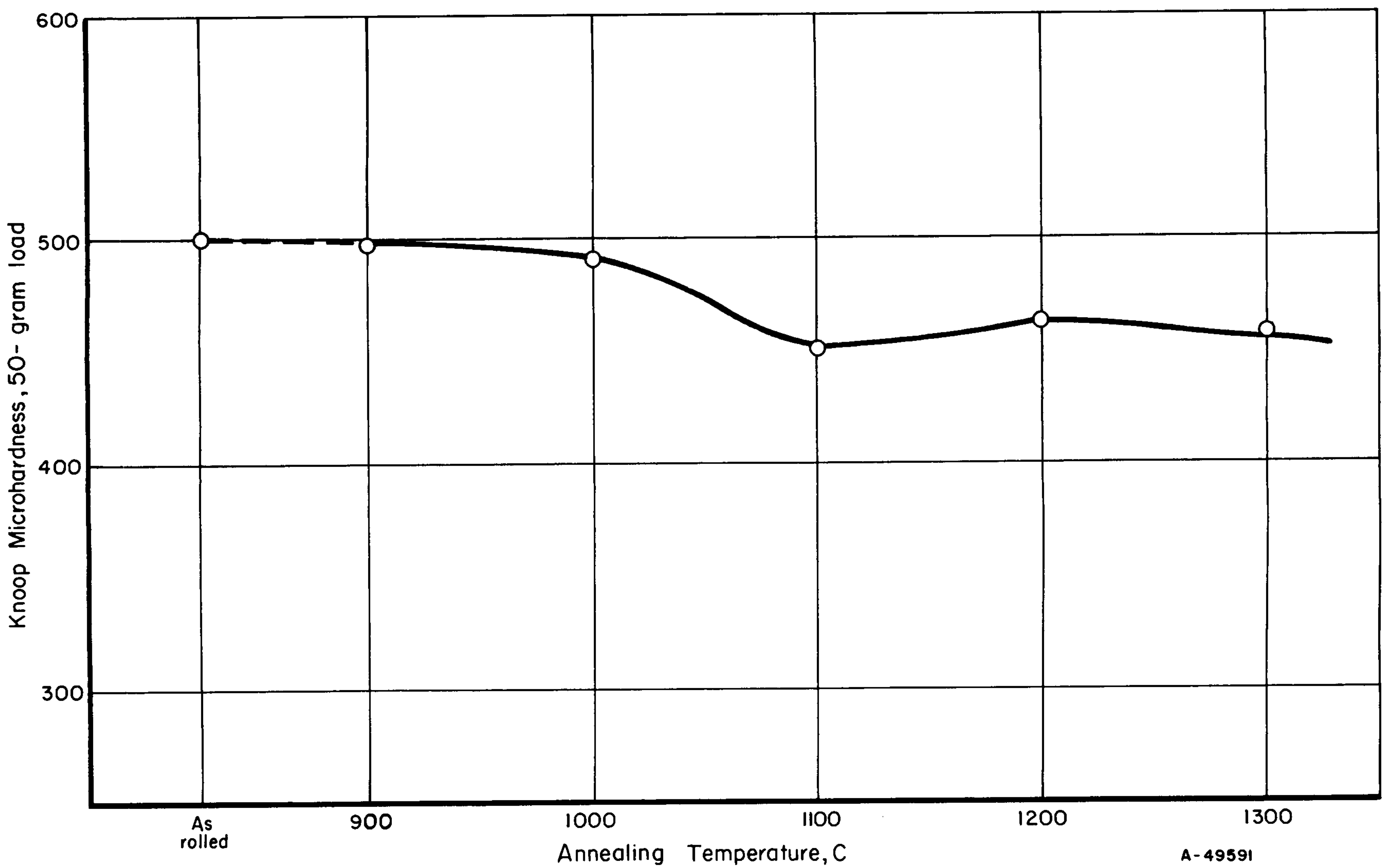
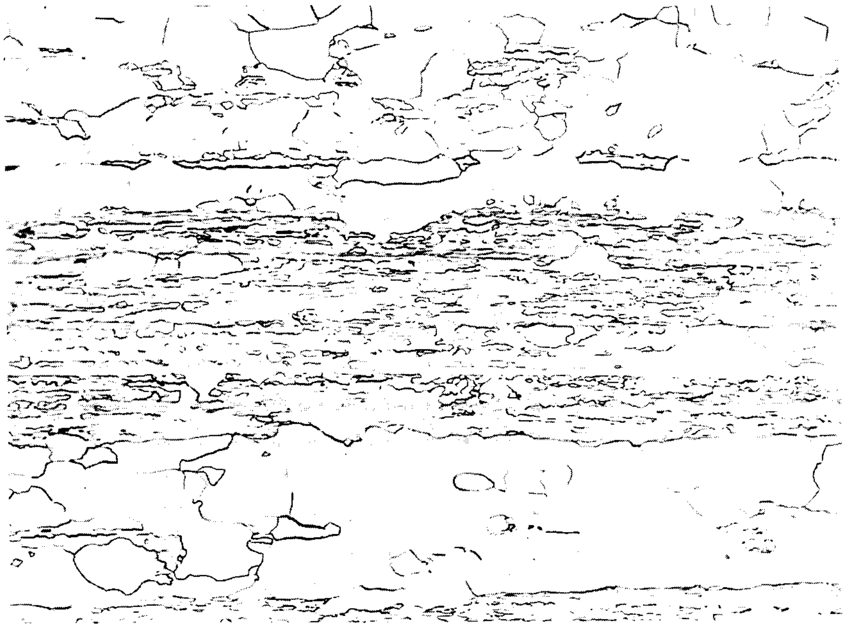


FIGURE 4. RELATIONSHIP BETWEEN MICROHARDNESS AND ANNEALING TEMPERATURE FOR THE COLUMBIUM-40 WT% VANADIUM ALLOY PREVIOUSLY ROLLED TO 5% REDUCTION IN THICKNESS

Ductility Improvement at Low Temperatures

The experiments were designed to investigate the low-temperature behavior of ductile columbium and brittle tungsten. It is known that twins formed in tungsten at low temperatures always have cracks associated with them. Therefore, it was considered that twins without cracks may be introduced at higher temperatures by shock loading. As a result, compression specimens of tungsten and columbium were explosively deformed at room temperature under a load of 100 and 90 kilobars, respectively. Figure 5 shows typical photomicrographs of the specimens before and after deformation. Mechanical twins are present in the columbium specimen but not in the tungsten specimen, while cleavage cracks are present in the columbium and extensive grain-boundary cracks are present in the tungsten. It appears, therefore, that the particular explosive deformation techniques as used at low temperatures cannot produce twins without cracks in either tungsten or columbium.



100X 15080

a. Recrystallized Tungsten.

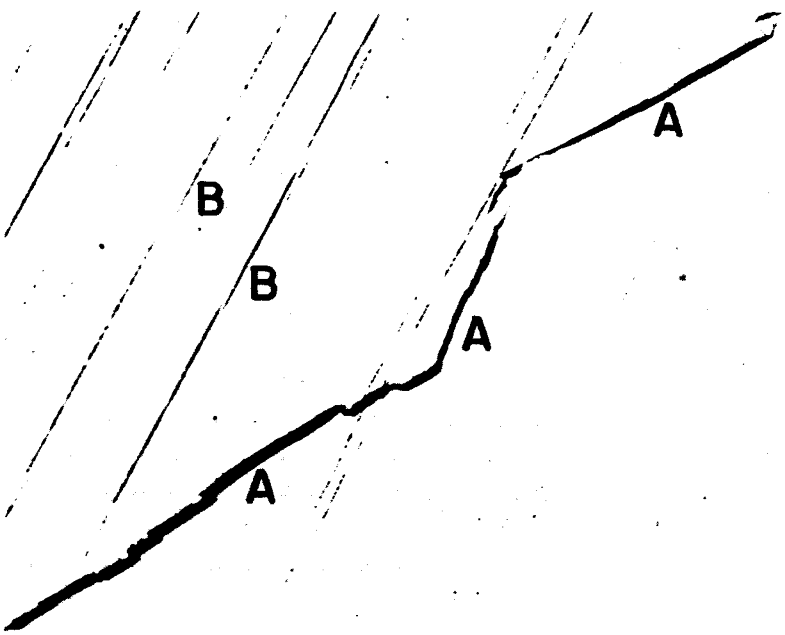
100X 15081

b. Fully Recrystallized Columbium.

FIGURE 5. PHOTOMICROGRAPHS SHOWING EFFECT OF COMPRESSIVE SHOCK LOADING ON TUNGSTEN AND COLUMBIUM

100X 15669

c. Tungsten Specimen in a. After Explosive Loading to 100 Kilobars. Note Grain-Boundary Cracks and Absence of Deformation Twins.



100X 15665

d. Columbium Specimen in b. After Explosive Loading to 90 Kilobars. Note Cleavage Cracks (A) and Deformation Twins (B).

FIGURE 5. (CONTINUED)

CURRENT AND FUTURE WORK

The current work is concentrated on measurements of the high-temperature strengthening effect in the columbium-40 wt % vanadium alloy. Tensile tests are being carried out on this material in three conditions: (1) as-annealed, prestrained at slip temperatures, (2) prestrained at twinning temperatures. After annealing specimens (a) and (b) at 1000 C for one hour to produce similar dislocation arrangements, all specimens will be tested in the temperature range 600 to 1000 C, at strain rates close to 10^{-3} sec^{-1}. At the same time, constant-load creep tests of a similar series of specimens are being carried out to investigate the magnitude of the twin hardening under creep conditions, since it is known that the creep resistance of this alloy is deficient.

In addition, the annealing characteristics of the columbium-40 wt % vanadium alloy in the slipped condition are being studied by metallographic and microhardness techniques for the purposes of comparison with the data already obtained with the twinned alloy.

REFERENCES

(1) Tardiff, H. P., Claisse, F., and Chollet, P., Response of Metals to High Velocity Deformation, Interscience Publishers (1961), p 389.

(2) Reid, C. N., Proposed Research Program to National Aeronautics and Space Administration, "Improvement of Creep Strength and Low-Temperature Ductility of Refractory Metals by Means of Mechanical Twinning" (June 4, 1964).

(3) Jaffee, R. I., Sims, C. T., and Harwood, J. J., "Proceedings of the Third Plausee Seminar", 1958, Reutte, Tyrol.

(4) Schnitzel, R. H., and Keith, G. H., J. of Less-Common Metals, 4, 98 (1962).

(5) McMahan, C. J., Sc. D. Thesis, M.I.T. (1963).

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Data reported are recorded in Battelle Laboratory Record Book No. 21653.

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